

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of Kazuhiro YANAGISAWA

Application No.: 10/595,084

Filed: February 3, 2006

For: RUBBER MASTER BATCH AND PRODUCTION METHOD THEREOF

Group Art Unit: 1796

Examiner: Angela C. Scott

Confirmation No.: 2328

DECLARATION UNDER 37 C.F.R. § 1.132

I, Fumiteru Nishiura, declare that:

I am a co-worker of Mr. Kazuhiro Yanagisawa and Mr. Kazuaki Someno who are the inventors of the above-captioned patent application.

I received my Master of Science and Technology from Keio University in 2003, and have been employed by Bridgestone Corporation since 2003, where I have been engaged mainly in research and development of new materials for a tire.

I have made the following experiments in order to measure a dispersibility of a filler in a rubber master batch, when a high shear mixer is used for preparing a filler slurry as taught by Yanagisawa et al. (US 2003/0088006), or when a rubber solution and a filler slurry are previously mixed and then the resulting mixture is introduced into a static mixer as suggested by Lopez-Serrano Ramos et al. (US 2002/0111413).

Experimental Procedure

<Comparative Example 2>

A rubber solution is prepared by diluting a field latex of a natural rubber (pH is adjusted to 10.5 with aqueous ammonia, a content of rubber component: 27.4 mass%) with deionized water so as to adjust the content of rubber component to 20 mass%. Also, carbon black N110 is added to deionized water, charged into a colloid mill [a diameter of a rotor: 50 mm, a clearance between a rotor and a stator: 0.7 mm] and passed therethrough once at a revolution number of 3000 rpm to prepare a filler

slurry. The rubber solution is charged at a flow rate of 500 mL/min and the filler slurry is simultaneously charged at a flow rate of 1000 mL/min into a tank provided with an impeller-type agitator for 5 minutes with agitating. After the completion of the charge, formic acid is added with agitating to adjust pH to about 5 to complete the coagulation. The resulting coagulated mass is collected with a screen having a mesh size of 30, washed with water and dried to obtain a rubber master batch.

#### <Comparative Example 3>

The rubber solution A and the filler slurry B described in the comparative example 1 of the present application are charged into a tank so that 50 parts by mass of the filler is mixed with 100 parts by mass of the rubber component and agitated. Then, the resulting mixture is charged into the static mixer described in the example 1 of the present application and mixed. Further, the mixture thus obtained is charged into the tank and then formic acid is added with agitating to adjust pH to about 5 to thereby complete the coagulation. The resulting coagulated mass is collected with the screen having the mesh size of 30, washed with water and dried to obtain a rubber master batch.

#### <Evaluation of homogeneity>

With respect to the resulting rubber master batches, the homogeneity is measured according to the method described in the specification of the present application. Results obtained from the above tests and the results described in the present specification are summarized in the following table.

Table: Homogeneity of the resulting rubber master batch

	Comparative Example 1	Comparative Example 2	Comparative Example 3	Example 1	Example 2
n=1	54.0	52.0	48.1	50.7	50.5
n=2	57.3	53.3	44.1	50.6	48.8
n=3	49.1	51.1	50.4	50.2	49.6
n=4	53.4	50.8	51.7	49.5	49.8
n=5	50.5	49.2	52.0	51.0	49.3
n=6	58.6	48.9	48.8	49.9	49.2
n=7	48.6	52.7	48.3	49.9	48.9
n=8	56.1	54.9	51.4	50.8	50.1
n=9	51.9	50.6	49.6	50.0	49.7
n=10	55.0	51.8	47.2	50.4	50.1
Average	53.5	51.5	49.2	50.3	49.6
$\sigma$	3.4	1.8	2.4	0.5	0.6

## (Summary)

The rubber master batches of the comparative examples 2 and 3 are poor in the homogeneity because the scattering in the amount of the filler compounded is large and the average amount of the filler compounded is far from a theoretical value, i.e. 50.

In the comparative example 2, the high shear mixer is not used in the mixing (coagulating) step of the rubber solution and the filler slurry, and thereby the coagulation progresses inhomogeneously to degrade the homogeneity of the rubber master batch.

In the comparative example 3, the rubber solution and the filler slurry are previously mixed and most of the mixture is inhomogeneously coagulated in the first mixing step. Moreover, the inhomogeneity in the rubber master batch cannot be eliminated by further charging the previously mixed mixture into the static mixer.

Consequently, it is confirmed that the use of the high shear mixer in the mixing step of the rubber solution and the filler slurry is superior in the homogeneity to the use of the high shear mixer in the preparing step of the filler slurry as taught by Yanagisawa et al. (US 2003/0088006). Further, it is confirmed that the simultaneous

injection of the rubber solution and the filler slurry into the static mixer is superior in the homogeneity to the injection of the mixture, which is prepared by previously mixing the rubber solution and the filler slurry, into the static mixer as suggested by Lopez-Serrano Ramos et al. (US 2002/0111413).

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under § 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: 7 / 15 / 2009 Declarant: Fumiteru Nishiura  
Fumiteru Nishiura